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"Alkaline Activation" as a procedure for the transformation of fly ashes into cementitious materials.

Part IV. Other applications

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Abstract

In general terms it is widely acknowledged that the alkali activation of fly ashes can produce a material with similar cementing features than those of Ordinary Portland Cement. Actually, the alkali activation of fly ashes is a singular procedure in which the dark grey powder originating from coal power plants is mixed with certain alkaline activators (alkaline dissolution); and then cured at a certain temperature to form solid hardened materials. The alkaline activation of fly ashes is consequently of great interest regarding the development of new and environmentally friendly binding materials with similar or superior properties to those of other well known binders.

The present paper discusses the fundamental technological aspects for producing high quality alkali cement using alkali activated fly ash as the main raw material. The resulting material exhibits a series of properties and characteristics of interest, including: high early age flexural and compressive strengths, rapid or slow setting, low drying shrinkage and so on. Due to their good technical properties and durability, as well as the ease with which these materials can be adapted (for manufacturing purposes) to existing facilities, these new cements are particularly suitable for: i) The precast industry; (ii) As protective coatings of materials with no capacity of fire resistance; (iii) Production of lightweight materials.

Keywords: fly ash, alkali activation, geopolymer, lightweight materials, fire protection

1 Introduction

The literature on alkali-activated systems has been growing steadily since the nineteen fifties, but the considerable store of information now available is not easily assimilated. The results have often been interpreted rather empirically, sometimes with little justification, while commercial interests have on occasion imposed restrictions on their dissemination. While fundamental studies have naturally been published, the comparison of findings is often hindered by differences in the experimental approach adopted between different research groups [**1-6**]. A more consistent picture of the chemistry underlying phase development and product performance has begun to develop only recently. In this context, the Spanish group authoring this paper (Eduardo Torroja Institute, CSIC) has accumulated considerable experience with these alkali-activated systems over the last 20 years and has contributed to the growing body of literature on the systematic characterisation of the chemical fundamentals of these cements [**7-13**].

The reaction of a solid aluminosilicate with a highly concentrated aqueous alkali hydroxide or silicate solution produces a synthetic alkaline aluminosilicate material [4-**7**]. The processing of these materials, which may perform comparably to traditional cementitious binders in a range of applications, emits significantly less greenhouse gas than Portland cement manufacture [**14**]. Depending on the raw materials and processing conditions used, alkali-activated binders may feature high compressive strength, low shrinkage, fast or slow setting, acid resistance, fire resistance and low thermal conductivity [**15-25**]. These binders should not obviously be regarded to be a panacea for all material selection problems, but rather a solution that, with suitable mix and processing design, may be tailored to optimise the properties and/or reduce the costs in materials for a given application. This article addresses several aspects of the intrinsic structure and properties of these binders (inorganic polymers) and a number of possible applications.

2 Background

Many articles have been published which focus on the evolution of mechanical strengths of alkali activated fly ash pastes and mortars. However, very few papers refer to the manufacture of concretes with alkali activated fly ash as the binder component [**26-30**].These papers show how the properties of alkali-activated fly ash concrete, like the characteristics of conventional concrete, are affected by a series of factors related to mix dosing and curing conditions. Contrary to conventional concrete, however, these new types of concrete can attain high strengths in very short times (1 day) and subsequently develop further strength at a slower rate.

In Figure 1(a) the values of compressive strength are shown for the OPC mortar at 25ºC and curing 12h. at 45ºC and for two different alkali activated fly ash mortar (without OPC) initially cured during 20h at 85ºC and activated with 8M NaOH solution. It is observed in this figure that the mortars of ash present a very high strength development in the course a few hours (higher than the OPC mortar in that same short space of time). In all cases, a slight and progressive evolution in the mechanical strengths was noted at later ages. Also, in Figure. 1(b) it can be observed that AAFA concrete presented very good compressive strengths at a very short curing time (6h. 26.7 MPa), this value can increase until 45.8 MPa after 20h. of curing.

Another important characteristic is shrinkage. Normally when a material is submitted to certain environmental conditions, it loses water and contracts. As shown in Figure 2 the alkali-activated ash mortars experience very small shrinkage upon drying; clearly lower than that of Portland cement mortars. This indicates very good volume stability, an extremely important property when designing precast items. This is especially the case with pre-stressed concrete, which benefits from very much lower pre-stress losses than usual, thanks to its practically non-existent shrinkage. Very good volume stability is a highly valuable property in the design of precast pieces.

The shrinkage of OPC and AAFA mortars was determined, according to the ASTM C 806-87 standard, under a laboratory environment. Portland cement mortars had a sand/binder ratio of 3/1 and a water/cement ratio of 0.5. Two sets of curing conditions were used: (a) 20h at 22ºC, laboratory standard curing conditions and (b) 20h at 45ºC, hot weather curing conditions. The sand/binder ratio used in the fly ash mortars were 2/1 and the alkaline solution (8M NaOH) /ash ratio was 0.4. These mortars were cured for 20h at 85ºC and 98% relative humidity, usual curing conditions for this type of material. After curing, the 2.5x2.5x23cm specimens were stored in the laboratory at 21ºC and approximately 50% relative humidity.

An in-depth understanding of the mechanisms that govern the formation of cementitious products during the alkali activation of fly ash is an obvious pre-requisite to establishing the inter-relationships between starting composition, curing conditions, nanostructure and properties. The knowledge accumulated in this regard to date has made it possible to tailor the composition of the alkaline activator and the curing conditions to the features sought in the end product. The most recent research findings have confirmed the following.

- Concretes made with these materials can be designed to reach compressive strength values of over 40 MPa after short thermal curing times [**7-13**].
- Concrete made with alkali-activated fly ash (with no OPC) performs as well as traditional concrete and even better in some respects, exhibiting less shrinkage and a stronger bond between the matrix and the reinforcing steel [**26-30**]..
- In addition to its excellent mechanical properties, the activated fly ash is particularly durable and highly resistant to aggressive acids, the aggregate-alkali reaction and fire [**15-25**].
- This family of materials is capable of immobilising toxic and hazardous substances very effectively [**31-35**].

3 Applications

The present paper focuses on three possible applications for alkali-activated fly ash in the production of materials with special properties, namely:

1- Precast industry, monoblock railway sleepers
2- fire-resistant coating
3- lightweight matrices in place of traditional cores in sandwich panels.

In all these applications, the materials generated by the alkaline activation of fly ash constitute a single family of composite materials with characteristics attributable to cement, fired clay and zeolites (such as rapid mechanical strength development, fire resistance, dimensional stability, acid resistance and excellent bonding to reinforcing steel).

3.1. Prestressed monoblock railway sleepers

The proposed use of alkali-activated fly ash concrete as the binder in the manufacture of railway sleepers is based on the considerations set out below.

i) Concrete railway sleepers are elements of a “manageable” size but extraordinarily complex, technologically speaking. Their design and production require materials that ensure strength, durability, good bonding to the reinforcement and volume stability. Activated fly ash concrete meets all these requirements.

ii) Conventional pre-casting processes usually call for accelerated thermal curing of the concrete. Optimal alkaline activation of fly ash is attained under similar thermal curing conditions. Consequently, production processes need not be substantially altered to accommodate the change from one raw material (Portland cement) to others (fly ash and alkaline activators).

iii) In pre-stressed elements, the steel reinforcement is pre-stressed before the concrete is cast in the mould. Given that alkali-activated fly ash concretes can develop very high early age strength (in the first 12-20 hours) and bond well to the reinforcement, the time the concrete must remain in the mould can be shortened. That, in turn, would lead to a significant rise in plant productivity.

iv) The difficulties encountered in attaining acceptable quality and durability in conventional concrete products subjected to accelerated curing cannot be readily surmounted today. The difficulty lies in the fact that the measures recommended in codes of good practice (such as the limitation of curing temperatures to 60 ºC or the use of aggregate that remains inert at high temperatures) are normally insufficient and nearly always incompatible or barely reconcilable with mass production. The use of activated ash could change the present scenario radically. Alkaline concrete made from activated ash can be expected to be highly durable, given its chemical-mineralogical similarity to certain natural and extraordinarily stable zeolites.

Finally, the economic and ecological issues involved also merit attention. Fly ash (which is being proposed as a total substitute for Portland cement in concrete manufacture) is an industrial by-product widely available on all six inhabited continents. It is generally found in stockpiles that occupy broad expanses of land because this waste is non-degradable.

Three preliminary trials were initially conducted at the plant. In each, two pre-stressed monoblock sleepers were made with activated fly ash concrete. A homogeneous mix was prepared in a small (125-litre) concrete mixer with materials dosed per cubic metre as shown in Table 1.

The specimens made for the first trial were kept in a mould at a temperature of 85 to 90 ºC for 20 hours. In the second trial, the activating solution was modified to improve the mechanical properties of the material. The third and last trial aimed to optimise concrete batching (further to the findings for the two previous trials) and curing conditions. To this end, the alkali activator concentrations were again modified and the flow of steam was changed slightly to raise the curing temperature to 110-115 ºC. The curing time was shortened to 10 hours.

Fifteen-centimetre cubic moulds were filled with the same concrete used to prepare the sleepers and cured under the same conditions to determine the mechanical strength of the material (see Figure 3).

The findings for these concretes, studied with a view to the manufacture of railway sleepers, showed that the material obtained developed high mechanical strength in a relatively short period of time (30 MPa after 6 hours at 95 ºC and 40 MPa after 10 hours at 115 ºC). Hence, as in Portland cement, curing temperature played a significant role in the results, although its effects differed, for the durability of this new cementitious material was unaffected by alterations in temperature. This constitutes an additional advantage to alkali-activated fly ash concretes from the perspective of standardised manufacturing and potentially higher productivity.

Another aspect that merits attention is the possibility of producing different qualities of concrete (measured in terms of mechanical strength, for instance) by modifying conditions such as the nature of the alkali activator used. Activators containing a certain amount of soluble silica are able to improve ash reactivity or even stimulate the formation of a more polymerized (silica-rich) and consequently more compact and mechanically stronger final product. This finding is of particular importance, for it means that materials, prices and other factors can be adapted to the needs of the precasting industry.

Finally, attention should be drawn to the adaptability of the material to the existing industrial facilities (the railroad sleepers studied were manufactured without significantly changing the conventional process). Moreover, this new material was used in sleeper manufacture because of the improvements afforded in product performance. Shrinkage, for instance, was negligible compared to the values recorded in Portland cement concrete, while an exceptionally good bond formed between the new material and the pre-stressed steel. While concrete sleepers made with Portland cement fail under a load of 46 tonnes (equivalent to a strength of 80 MPa in cubic specimens), these activated fly ash concrete sleepers withstood loads of up to 50 tonnes.

Figure 4 shows photographs of industrial scale monoblock railway sleeper manufacture using the trial 3 dosage (see Table 1). The railway sleepers were manufactured with no need to significantly alter the manufacturing stream in place at the plant. The most notable change affected curing temperature, raised from 50 to 85-90 ºC, which entailed building a pressurised steam injection tunnel (see Figure 4).

The sleepers were subjected to the mandatory static and dynamic tests. The values obtained met both Spanish and European code requirements. The ***static test*** consisted of applying a static load to the section studied to determine the load at first cracking. The ***dynamic test*** consisted of applying a steadily rising pulsating load to simulate exceptional impact and determine, as in the static test, the loads under which characteristic cracking appeared. The findings are given in Table 2.

3.2 Fire-resistant coating

The primary purpose in studying this particular application was to establish the fundamentals for developing a series of new alkali-activated fly ash-based inorganic materials usable as a fire-resistant coating for fibreglass or carbon fibre matrices.

Despite their lower density, sheets of fibreglass- or carbon fibre-reinforced polymeric resin can develop strength similar to the values characteristic of steel. The main drawback to such materials is their low resistance to high temperatures and their combustibility when directly exposed to a flame. The Figure 5 show that fibreglass and carbon fibre deteriorate after 8 to 20 seconds of direct exposure to a flame.

The interaction between fire and these materials can be essentially described in terms of the following two factors.

Reaction to fire is the relative capacity of materials to favour the initiation or propagation of a fire, i.e., their contribution to fire as laid down in the existing legislation [to determine a material's reaction to fire, its ignitability is tested "by direct impingement of flame"]. The procedure consists of holding the sample in a vertical position in a Bunsen burner flame (Spanish, European and international standard UNE-EN-ISO 11925-2).

Fire resistance (or fire rating) is the capacity of a material to retain its bearing capacity, integrity and stability for a given amount of time (a property generally required in structural members).

These two factors (**reaction to fire** and **fire resistance**) should always be considered separately, because a product that reacts well to fire may not be fire-resistant. This is the case of self-extinguishing products, for instance (inasmuch as they generate no flame they meet reaction to fire requirements, but since they are deteriorated and consumed in the process, they fail to remain intact long enough to comply with fire resistance provisions).

In the present study, these glass or carbon fibre-reinforced resin specimens were coated with an alkali-activated fly ash slurry prepared with different alkaline activators in the presence or absence of some extra fillers. The effect of the thickness of the layer of slurry was also studied.

These materials were exposed directly to a flame as specified in standard UNE-EN-ISO 11925-2: the coated samples were placed underneath an extractor hood and held with tongs in a vertical position in contact with a Bunsen burner flame.

The main findings are summarised in Figure 5 (parts c and d).

-***Material 1*** was a carbon fibre-reinforced resin coated with a 6M NaOH-activated fly ash slurry**.** The photo in Figure 5c shows the specimen after the test, which lasted 8 minutes. At longer times, the sample first began to give off an odour and then to smoke profusely, but the flame was not propagated.

-Material 2 was coated with 80 % fly ash + 20 % inorganic additions + activator. In this case the relatively small proportion of inorganic materials added to the mix lengthened fire resistance considerably: to 40 minutes where the resin was slurry-coated. The coating was 3 to 5 mm thick. The flame was not propagated to the rest of the specimen in this case either (see Figure 5d).

In light of the results with material 2 (where a thicker layer improved fire resistance substantially), the other materials were also tested to determine whether several layers of slurry would enhance fire resistance. This hypothesis was not confirmed, however, probably because applying more than one layer of paste to the substrate greatly reduced bonding effectiveness.

This study showed that the improvements achieved with an alkali-activated fly ash or alkaline inorganic polymer coating included the following.

-The primary advantage obtained with the use of this type of coating is non-propagation of the flame.

-The resin ignites where the flame is applied but the flame is not propagated upward. The flame disappears when the resin is consumed.

-The most promising results were obtained with material 3, probably because the presence of a extra filler afforded a better bond between the slurry and the substrate, while also lowering the internal stress in the slurry itself.

3.3 Lightweight matrices in place of traditional cores in sandwich panels.

All materials play an important role in construction today. Hence the future of the construction industry is highly conditioned by the development of new materials and the use of new construction methods, as well as by the application of building codes with increasingly demanding structural safety, fire protection, and acoustic and thermal insulation provisions. These new requirements can be difficult to meet with conventional metals, fired clay products, polymers and concrete.

The development of composites or materials containing a combination of different types of elements, and consequently exhibiting the full range of their respective properties, has acquired considerable importance in recent years in an attempt to meet these new needs.

Generally speaking, a composite is defined to be a multi-phase material that conserves a significant portion of the properties of its constituent phases, which are combined as optimally as possible. Further to the principle of combined action, the properties sought are best obtained when a composite containing two or more materials is designed on the grounds of the scientific information available.

Most composites comprise two phases, a continuous matrix and a disperse phase that surrounds or envelops the matrix.

Sandwich panels are presently manufactured for any number of applications. As a rule, they provide acoustic and thermal insulation. The success of these panels can be attributed primarily to the following properties:

- high load bearing capacity, light weight
- excellent and durable thermal insulation
- good heat and humidity barrier
- excellent water repellence
- weather- and aggressive environment-resistant skins
- rapid installation with no need for complex hoisting equipment
- economical production
- fire resistance in panels with mineral wool cores.

Standard sandwich panels are three-layered. The rigid surfaces (skins), which have a fairly high modulus of elasticity, are held apart by a thick, lightweight core with sufficient bending stiffness to withstand most stress. The core also serves as thermal insulation. Each component is relatively weak and flexible taken by itself, but the combined panel comprises a rigid, strong, lightweight structure.

A number of materials may be used for the skin: fibreglass- and carbon fibre-reinforced epoxy resin (the resins are usually unsaturated polyesters and vinyl esters). Other materials commonly used include wood, unreinforced polymers and metal (normally galvanised sheet steel and aluminium or, for specific purposes, copper). The surface elements range from 0.25 to 12.7 mm thick.

The core has two structural functions. First, it separates the two skins and resists deformation perpendicular to the plane of these two surfaces. And second, it affords some degree of shear strength along the planes perpendicular to the skin.

The core may consist of either organic or inorganic materials. The organic materials most often used include rigid expanded polyurethane and extruded polystyrene. The drawback to both these materials is that they generate toxic fumes when ignited. Organic matrices cannot therefore generally be used for applications in which temperatures of over 200 ºC are reached.

The most common inorganic cores include mineral wool and fibreglass, both of which are non-flammable and chemically inert. The drawback is that they cannot withstand loads. Other possible materials include synthetic rubber and cement.

The present study explored the possibility of manufacturing these cores with alkali-activated fly ash, using a series of air-entraining admixtures to lower the density of this material. The procedure followed is described below.

Three air-entraining admixtures, A, B and C, were initially tested at a dosage of 1 %wt (see Figure 6). In all three cases the activating solution used was an 8-M NaOH (85 %wt) and Wg (15 %wt) mix, denominated N8W15. The pastes were mixed with the air-entraining product and poured into a mould holding three 4x4x16-cm prismatic specimens. The material was subsequently cured in a kiln for 20 hours at 85 ºC and very high humidity. After removal from the mould the next day, the specimens were tested for density and mechanical strength.

Admixture A led to segregation, i.e., it generated porosity on the surface only, and was consequently ruled out. Admixture B yielded a material with a density of 1 000 kg/m^3 and compressive strength values of 7.2 ±2.7 MPa (see Figure 6). This admixture reacted very rapidly with the paste, however, reducing its workability and causing homogenisation problems. The material obtained with admixture

C, by contrast, had evenly distributed pores, a density of 603.5 kg/m^3 and a compressive strength of 2.0±0.11 MPa.

Manufacture of large (40 x 40 x 6-cm) core panels was undertaken with admixture C. Here also the paste was poured into a mould and cured in a kiln at 85 ºC for 1 hour. When a suitable consistency was reached, it was floated and returned to the kiln for a further 19 hours. Figure 7 depicts the mould used and the material generated.

Subsequent studies were conducted to optimise the percentage of sodium silicate added to the alkaline solution and the dosage of air-entraining admixture. The 40x40x6-cm panels, obtained with solution DW15 [NaOH (85 %) + waterglass (15 %)] and 1.2 % (by fly ash mass) admixture, exhibited a density of ≈400-450 kg/m^3 and compressive strength of ≈2 MPa.

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Table 1. Concrete dosage used to manufacture the sleepers

Material (m^3)	TEST Nº 1	TEST Nº 2	TEST Nº 3
FLY ASH	465 Kg	465 Kg	465 Kg
SILICA SAND	825 Kg	825 Kg	825 Kg
AGGREGATE	1050 Kg	1050 Kg	1050 Kg
ACTIVATOR	NaOH [Na_2O]=8,7%	NaOH + W	NaOH +W
LIQ/SOLID RATIO	0.37	0.50	0.52
Curing Conditions	20 h, 85 ºC	20 h, 85 ºC	10 h, 110 ºC

Table 2. Results for monoblock railway sleepers made with Portland cement-free, alkali-activated fly ash concretes

		Code value		AAFA sleeper
		RENFE*	UIC**	
Static test	**1st crack**	13 t	15 t	42.57 t
	Failure	28.6 t	33 t	59.47 t
Dynamic test	**1st crack > 0.05 mm**	195 kN	228 kN	350 kN
	Failure	286 kN	334 kN	390 kN

* Spanish national railway system ** International Europe Union of Railways

Figure 1. Compressive mechanical strengths of (a) mortars of OPC and AAFA and (b) AAFA concrete. The AAFA mortars and concrete were activated with NaOH 8M, curing until 20h. at 85ªC.

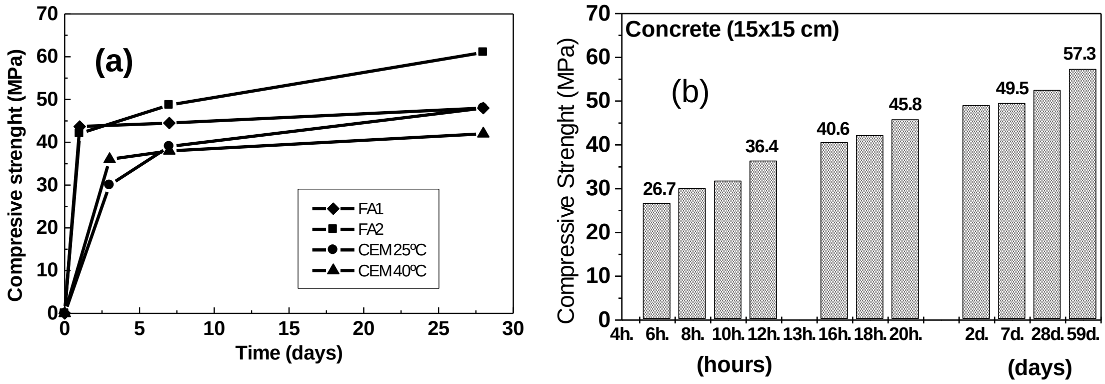

Figure 2. Shrinkage on drying in cement and alkali-activated fly ash mortars **(**Test conducted on 2.5x2.5x23-cm mortar specimens. (ASTM C 806-87).

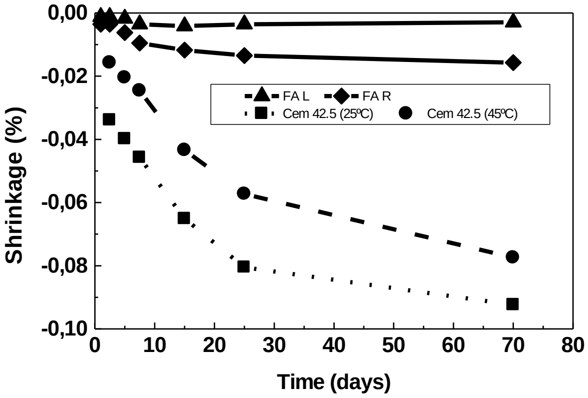

Figure 3. Mechanical strength of concrete specimens found in trials 1, 2 and 3.

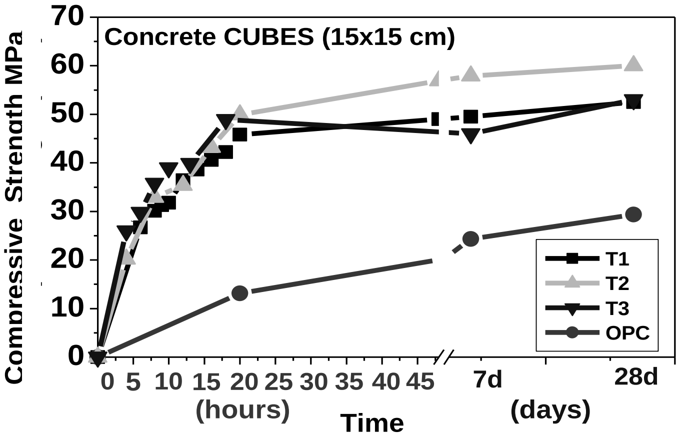

Figure. 4. Photographs of the industrial scale manufacture of monoblock railway sleepers with (OPC-free) alkali-activated fly ash concrete

Figure 5. Photographs of fire protection test. (a) Fibreglass + epoxy resin, exposure time 8s; (b) carbon fibre + epoxy resin, exposure time 20s.; (c) coating *Material 1* exposure time 8m ; (d) coating *Material 2* exposure time 40m.

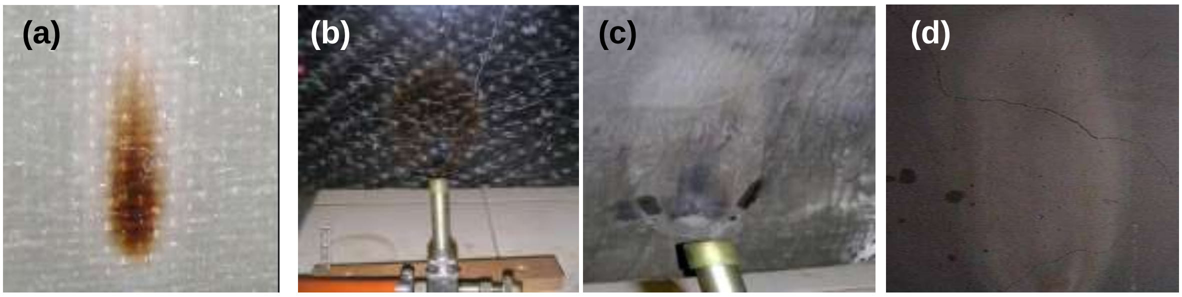

Figure 6. Lightweight materials

Admixture A	**Admixture B**	**Admixture C**
		
Segregation Surface porosity Poor homogenisation	Very rapid, uncontrolled reaction Homogenisation problems	Reaction after 1 hour Evenly distributed pores

Figure 7. Physical appearance of the panels manufactured